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# 4-Chlorobutyl 7-chloro-1-cyclopropyl-4-(1,3-diethyl-4,6-dioxo-2-sulfanylidene-1,3-diazinan-5-ylidene)-6-fluoro-1,4-dihydroquinoline-3-carboxylate

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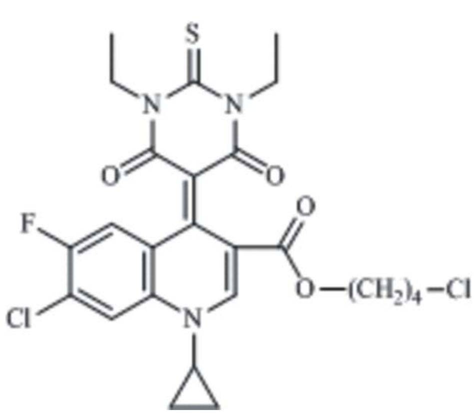
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.007 Å; $R$ factor = 0.078; $wR$ factor = 0.230; data-to-parameter ratio = 14.4.

The title compound, $C_{25}H_{26}Cl_2FN_3O_4S$, contains two bioactive moieties (thiobarbituric acid and fluoroquinolone). In the crystal, molecules are linked *via* C—H$\cdots$O and C—H$\cdots$F hydrogen bonds, forming two-dimensional slab-like networks lying parallel to the *bc* plane. The benzene ring substituted by F and Cl atoms and the 4-chlorobutyl group seem to be partly disordered, however attempts to model the disorder were unsuccessful.

## Related literature

For the biological activity of fluoroquinolone derivatives, see: Li *et al.* (2000); Baker *et al.* (2004); Mitscher (2005). For the crystal structures of some fluoroquinolone and 1,3-diethyl-2-thiobarbituric acid derivatives, see: Al-Qawasmeh (2012); Sweidan *et al.* (2012); Shishkin *et al.* (1997).



## Experimental

### *Crystal data*

$C_{25}H_{26}Cl_2FN_3O_4S$
$M_r$ = 554.45
Monoclinic, $C2/c$
$a$ = 24.3035 (15) Å
$b$ = 13.8310 (8) Å
$c$ = 16.4507 (16) Å
$\beta$ = 107.345 (8)°
$V$ = 5278.3 (7) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.37 mm$^{-1}$
$T$ = 293 K
0.30 × 0.15 × 0.06 mm

### *Data collection*

Agilent Xcalibur Eos diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min}$ = 0.606, $T_{max}$ = 1.000
12511 measured reflections
4664 independent reflections
2477 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.047

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.230
$S$ = 1.02
4664 reflections
325 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.58 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.40 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H$\cdots$$A$ | $D$—H | H$\cdots$$A$ | $D\cdots A$ | $D$—H$\cdots$$A$ |
|---|---|---|---|---|
| C18—H18A$\cdots$O2$^{i}$ | 0.98 | 2.32 | 3.014 (6) | 127 |
| C20—H20A$\cdots$O3$^{ii}$ | 0.97 | 2.56 | 3.233 (6) | 126 |
| C25—H25A$\cdots$F1$^{iii}$ | 0.97 | 2.51 | 3.371 (10) | 148 |

Symmetry codes: (i) $-x, y, -z+\frac{1}{2}$; (ii) $-x, -y+1, -z$; (iii) $x, y+1, z$.

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2478).

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# supplementary materials

*Acta Cryst.* (2013). E**69**, o1191 [doi:10.1107/S1600536813016024]

## 4-Chlorobutyl 7-chloro-1-cyclopropyl-4-(1,3-diethyl-4,6-dioxo-2-sulfanylidene-1,3-diazinan-5-ylidene)-6-fluoro-1,4-dihydroquinoline-3-carboxylate

**Kamal Sweidan, Salim F. Haddad, Murad A. AlDamen and Ahmed Al-Sheikhb**

### Comment

A vast number of fluoroquinolones (Baker *et al.*, 2004) such as ciprofloxacin and moxifloxacin have therapeutic efficacy as anti-infective agents (Li *et al.*, 2000; Mitscher, 2005). The prepared molecule contains two bio-active moieties (thiobarbituric acid and fluoroquinolone), which may have a high impact biological effect.

From the X-ray structural analysis, it is interesting to note that this structure presents some zwitterionic character, since the bond length for C3—C10 is 1.449 (5) Å, intermediate between carbon-carbon single bond and double bond, while bond angles around C10 are close to 120°: C13—C10—C11 = 119.8 (4), C11—C10—C3 = 120.5 (4), and C13—C10—C3 = 119.5 (4)°. The crystal structure reveals the negative charge to be localized at the thiobarbituric-acid ring, with a contribution of the enolate form [C10—C11 = 1.418 (6), C10—C13 = 1.406 (6), C11—O1 = 1.231 (5), C13—O2 = 1.229 (5) Å; see Sweidan *et al.*, 2012]. The carbon-sulfur bond length is 1.665 (5) Å, which is close to that observed in a 2-thioxo-1,2,3,4-tetrahydropyrimidine derivative [1.673 (9) Å, Shishkin *et al.*, 1997; see also Al-Qawasmeh, 2012].

The displacement parameters in the 4-chlorobutyl branch are rather high, indicating, together with residual electron density in the vicinity, some degree of disorder. For example, C22—C23 bond length, 1.402 (9) Å, is a bit short for a single C—C bond. After convergence, it seems that benzene ring substituted by F1 and Cl1 could also be partly disordered. However, attempts to model such disordered parts did not improve the picture.

### Experimental

The title compound was prepared as followed: 8 ml of thionyl chloride was added to a solution containing 1.5 g (5.3 mmol) of 7-chloro-1-cyclopropyl-6-fluoro-4-oxo-1,4-dihydroquinoline-3-carboxylic acid in 15 ml of dry THF. The resulting solution was refluxed for 6 h, then cooled, and evaporated under reduced pressure to remove the excess thionyl chloride. To the resulting residue, a solution of 1,3-diethyl-2-thiobarbituric acid (1.19 g, 6.0 mmol) and 1.5 ml (10.0 mmol) of dry triethylamine in 20 ml of dry THF were added, at room temperature. After stirring overnight, the resulting precipitate was filtered off, washed with $H_2O$/THF (1:2), and dried under reduced pressure. Yield: 1.5 g (51%). This solid was recrystallized from dichloromethane/diethylether, affording brownish crystals. A flat elongated crystal was mounted and data collected using five omega scans and a total of 277 frames with an exposure time of 76 s per frame.

### Refinement

All non-H atoms were refined anisotropically. H atoms were positioned geometrically, with C—H = 0.93 (aromatic CH), 0.96 (methyl $CH_3$), 0.97 (methylene $CH_2$), or 0.98 Å (methine CH), and constrained to ride on their parent atoms, with $U_{iso}$(H) = 1.5$U_{eq}$(carrier atoms) for methyl groups and 1.2$U_{eq}$(carrier atoms) otherwise. Highest difference peak and hole in the last difference map are 0.58 and -0.40 e/Å$^3$, respectively.

### Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

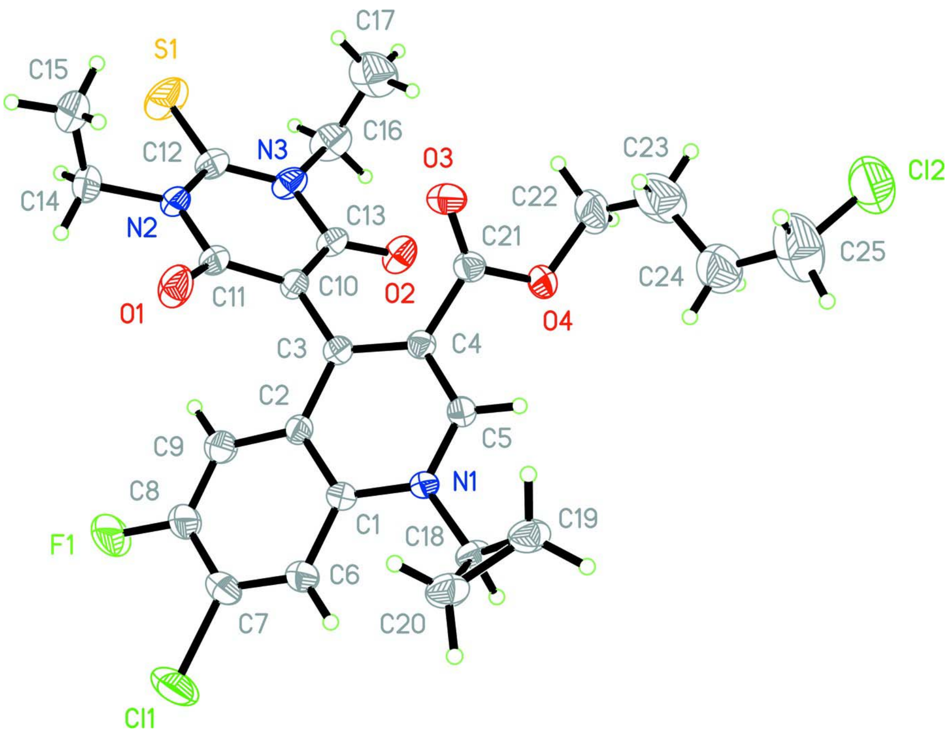


**Figure 1**

*ORTEP* drawing showing the molecular conformation of the title compound. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level. Hydrogen atoms are represented as small spheres of arbitrary radii.

### 4-Chlorobutyl 7-chloro-1-cyclopropyl-4-(1,3-diethyl-4,6-dioxo-2-sulfanylidene-1,3-diazinan-5-ylidene)-6-fluoro-1,4-dihydroquinoline-3-carboxylate

*Crystal data*

| | |
|---|---|
| $C_{25}H_{26}Cl_2FN_3O_4S$ | $F(000) = 2304$ |
| $M_r = 554.45$ | $D_x = 1.395$ Mg m$^{-3}$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å |
| Hall symbol: -C 2yc | Cell parameters from 2598 reflections |
| $a = 24.3035$ (15) Å | $\theta = 2.9$–$29.0°$ |
| $b = 13.8310$ (8) Å | $\mu = 0.37$ mm$^{-1}$ |
| $c = 16.4507$ (16) Å | $T = 293$ K |
| $\beta = 107.345$ (8)° | Parallelpiped, orange |
| $V = 5278.3$ (7) Å$^3$ | 0.30 × 0.15 × 0.06 mm |
| $Z = 8$ | |

*Data collection*

Agilent Xcalibur Eos diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0534 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min}$ = 0.606, $T_{max}$ = 1.000
12511 measured reflections
4664 independent reflections
2477 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.047
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.9°
$h = -28 \rightarrow 22$
$k = -16 \rightarrow 16$
$l = -19 \rightarrow 18$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.230
$S$ = 1.02
4664 reflections
325 parameters
0 restraints
0 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1011P)^2 + 5.5588P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.58 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.40 e Å$^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.24101 (6) | 0.17120 (14) | 0.14821 (12) | 0.0943 (6) |
| Cl1 | −0.19348 (8) | 0.14374 (13) | 0.09519 (18) | 0.1377 (10) |
| Cl2 | 0.08427 (9) | 1.00624 (14) | 0.18342 (15) | 0.1267 (8) |
| F1 | −0.07857 (15) | 0.0682 (2) | 0.1166 (3) | 0.1128 (14) |
| O1 | 0.03572 (14) | 0.2291 (3) | 0.0014 (2) | 0.0712 (11) |
| O2 | 0.12679 (13) | 0.4217 (2) | 0.2354 (2) | 0.0586 (9) |
| O3 | 0.08993 (13) | 0.5366 (2) | 0.0784 (2) | 0.0578 (9) |
| O4 | 0.06525 (13) | 0.6089 (2) | 0.1858 (2) | 0.0612 (10) |
| N1 | −0.08816 (13) | 0.4624 (2) | 0.1034 (2) | 0.0369 (8) |
| N2 | 0.12960 (16) | 0.2031 (3) | 0.0780 (2) | 0.0483 (10) |
| N3 | 0.17566 (15) | 0.3066 (3) | 0.1881 (2) | 0.0531 (11) |
| C1 | −0.08558 (17) | 0.3631 (3) | 0.1035 (3) | 0.0405 (10) |
| C2 | −0.03227 (17) | 0.3163 (3) | 0.1127 (3) | 0.0390 (10) |
| C3 | 0.01920 (16) | 0.3715 (3) | 0.1196 (2) | 0.0374 (10) |
| C4 | 0.01378 (17) | 0.4713 (3) | 0.1244 (3) | 0.0377 (10) |
| C5 | −0.03978 (18) | 0.5125 (3) | 0.1162 (3) | 0.0401 (10) |
| H5A | −0.0418 | 0.5794 | 0.1200 | 0.048* |
| C6 | −0.1360 (2) | 0.3092 (3) | 0.0963 (3) | 0.0552 (13) |
| H6A | −0.1710 | 0.3405 | 0.0887 | 0.066* |
| C7 | −0.1331 (2) | 0.2110 (4) | 0.1008 (4) | 0.0736 (17) |
| C8 | −0.0802 (2) | 0.1650 (4) | 0.1120 (4) | 0.0724 (17) |
| C9 | −0.0314 (2) | 0.2151 (3) | 0.1193 (3) | 0.0582 (14) |
| H9A | 0.0033 | 0.1821 | 0.1287 | 0.070* |
| C10 | 0.07331 (17) | 0.3246 (3) | 0.1239 (3) | 0.0401 (10) |
| C11 | 0.07575 (18) | 0.2519 (3) | 0.0643 (3) | 0.0462 (11) |

| | | | | |
|---|---|---|---|---|
| C12 | 0.1791 (2) | 0.2288 (4) | 0.1375 (3) | 0.0516 (12) |
| C13 | 0.12424 (17) | 0.3552 (3) | 0.1849 (3) | 0.0434 (11) |
| C14 | 0.1290 (2) | 0.1194 (4) | 0.0207 (4) | 0.0677 (15) |
| H14A | 0.1561 | 0.0711 | 0.0517 | 0.081* |
| H14B | 0.0909 | 0.0905 | 0.0041 | 0.081* |
| C15 | 0.1443 (3) | 0.1470 (4) | −0.0569 (4) | 0.092 (2) |
| H15A | 0.1433 | 0.0907 | −0.0915 | 0.138* |
| H15B | 0.1823 | 0.1743 | −0.0409 | 0.138* |
| H15C | 0.1171 | 0.1938 | −0.0886 | 0.138* |
| C16 | 0.2289 (2) | 0.3434 (5) | 0.2500 (4) | 0.0736 (17) |
| H16A | 0.2564 | 0.2910 | 0.2675 | 0.088* |
| H16B | 0.2200 | 0.3671 | 0.3001 | 0.088* |
| C17 | 0.2549 (3) | 0.4222 (6) | 0.2130 (5) | 0.107 (2) |
| H17A | 0.2893 | 0.4446 | 0.2545 | 0.161* |
| H17B | 0.2279 | 0.4746 | 0.1965 | 0.161* |
| H17C | 0.2643 | 0.3985 | 0.1639 | 0.161* |
| C18 | −0.14249 (17) | 0.5130 (3) | 0.0928 (3) | 0.0437 (11) |
| H18A | −0.1549 | 0.5188 | 0.1441 | 0.052* |
| C19 | −0.1571 (2) | 0.5954 (4) | 0.0329 (3) | 0.0594 (14) |
| H19A | −0.1768 | 0.6502 | 0.0484 | 0.071* |
| H19B | −0.1302 | 0.6116 | 0.0016 | 0.071* |
| C20 | −0.1889 (2) | 0.5045 (4) | 0.0120 (3) | 0.0610 (14) |
| H20A | −0.1817 | 0.4647 | −0.0324 | 0.073* |
| H20B | −0.2283 | 0.5032 | 0.0144 | 0.073* |
| C21 | 0.06135 (18) | 0.5399 (3) | 0.1271 (3) | 0.0455 (11) |
| C22 | 0.1124 (3) | 0.6771 (5) | 0.1981 (5) | 0.094 (2) |
| H22A | 0.1302 | 0.6866 | 0.2587 | 0.113* |
| H22B | 0.1412 | 0.6496 | 0.1749 | 0.113* |
| C23 | 0.0952 (3) | 0.7671 (6) | 0.1597 (6) | 0.130 (3) |
| H23A | 0.0825 | 0.7578 | 0.0985 | 0.156* |
| H23B | 0.1288 | 0.8087 | 0.1728 | 0.156* |
| C24 | 0.0490 (3) | 0.8188 (5) | 0.1840 (6) | 0.112 (2) |
| H24A | 0.0610 | 0.8283 | 0.2452 | 0.134* |
| H24B | 0.0146 | 0.7789 | 0.1694 | 0.134* |
| C25 | 0.0342 (4) | 0.9178 (6) | 0.1395 (6) | 0.145 (4) |
| H25A | −0.0032 | 0.9385 | 0.1429 | 0.174* |
| H25B | 0.0314 | 0.9106 | 0.0798 | 0.174* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0580 (9) | 0.1077 (14) | 0.1167 (15) | 0.0380 (9) | 0.0255 (9) | −0.0067 (10) |
| Cl1 | 0.0780 (11) | 0.0645 (11) | 0.284 (3) | −0.0280 (9) | 0.0751 (15) | −0.0082 (14) |
| Cl2 | 0.1264 (17) | 0.0849 (14) | 0.161 (2) | −0.0058 (12) | 0.0313 (15) | 0.0051 (12) |
| F1 | 0.094 (3) | 0.0357 (18) | 0.222 (5) | −0.0059 (17) | 0.067 (3) | 0.003 (2) |
| O1 | 0.052 (2) | 0.082 (3) | 0.072 (3) | 0.0024 (19) | 0.0056 (19) | −0.036 (2) |
| O2 | 0.0491 (19) | 0.075 (2) | 0.043 (2) | 0.0087 (17) | 0.0004 (16) | −0.0185 (17) |
| O3 | 0.0477 (18) | 0.070 (2) | 0.060 (2) | −0.0072 (17) | 0.0225 (17) | −0.0007 (17) |
| O4 | 0.0503 (19) | 0.046 (2) | 0.090 (3) | −0.0131 (16) | 0.0254 (19) | −0.0231 (18) |
| N1 | 0.0313 (18) | 0.036 (2) | 0.045 (2) | 0.0007 (16) | 0.0143 (16) | −0.0002 (16) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N2 | 0.048 (2) | 0.042 (2) | 0.061 (3) | 0.0062 (18) | 0.027 (2) | −0.0024 (18) |
| N3 | 0.035 (2) | 0.073 (3) | 0.047 (2) | 0.0094 (19) | 0.0070 (18) | −0.008 (2) |
| C1 | 0.038 (2) | 0.039 (3) | 0.046 (3) | −0.003 (2) | 0.017 (2) | −0.002 (2) |
| C2 | 0.038 (2) | 0.037 (2) | 0.043 (3) | 0.003 (2) | 0.012 (2) | −0.0028 (19) |
| C3 | 0.035 (2) | 0.042 (3) | 0.035 (2) | 0.001 (2) | 0.0089 (19) | −0.0015 (18) |
| C4 | 0.036 (2) | 0.040 (3) | 0.038 (3) | −0.001 (2) | 0.0111 (19) | −0.0021 (18) |
| C5 | 0.043 (2) | 0.037 (2) | 0.042 (3) | −0.002 (2) | 0.015 (2) | −0.0022 (19) |
| C6 | 0.042 (3) | 0.046 (3) | 0.080 (4) | −0.003 (2) | 0.022 (3) | −0.003 (2) |
| C7 | 0.057 (3) | 0.051 (3) | 0.120 (5) | −0.016 (3) | 0.036 (3) | −0.009 (3) |
| C8 | 0.065 (4) | 0.036 (3) | 0.126 (5) | 0.000 (3) | 0.044 (4) | 0.000 (3) |
| C9 | 0.051 (3) | 0.042 (3) | 0.086 (4) | 0.005 (2) | 0.027 (3) | 0.004 (2) |
| C10 | 0.038 (2) | 0.041 (3) | 0.041 (3) | 0.003 (2) | 0.012 (2) | −0.0051 (19) |
| C11 | 0.039 (2) | 0.046 (3) | 0.056 (3) | 0.003 (2) | 0.018 (2) | −0.005 (2) |
| C12 | 0.046 (3) | 0.059 (3) | 0.052 (3) | 0.009 (2) | 0.018 (2) | 0.003 (2) |
| C13 | 0.037 (2) | 0.056 (3) | 0.037 (3) | 0.005 (2) | 0.011 (2) | −0.004 (2) |
| C14 | 0.069 (3) | 0.049 (3) | 0.092 (4) | −0.001 (3) | 0.035 (3) | −0.021 (3) |
| C15 | 0.118 (5) | 0.074 (4) | 0.106 (5) | −0.016 (4) | 0.066 (4) | −0.034 (4) |
| C16 | 0.044 (3) | 0.108 (5) | 0.057 (4) | 0.010 (3) | −0.003 (3) | −0.010 (3) |
| C17 | 0.070 (4) | 0.130 (6) | 0.105 (6) | −0.020 (4) | 0.001 (4) | −0.013 (5) |
| C18 | 0.035 (2) | 0.050 (3) | 0.049 (3) | 0.008 (2) | 0.017 (2) | 0.006 (2) |
| C19 | 0.046 (3) | 0.060 (3) | 0.077 (4) | 0.014 (3) | 0.026 (3) | 0.024 (3) |
| C20 | 0.038 (3) | 0.073 (4) | 0.071 (4) | 0.010 (3) | 0.014 (3) | 0.005 (3) |
| C21 | 0.037 (2) | 0.047 (3) | 0.051 (3) | −0.001 (2) | 0.011 (2) | 0.000 (2) |
| C22 | 0.067 (4) | 0.065 (4) | 0.145 (7) | −0.019 (3) | 0.023 (4) | −0.037 (4) |
| C23 | 0.068 (5) | 0.105 (6) | 0.203 (9) | −0.009 (5) | 0.019 (5) | 0.021 (6) |
| C24 | 0.088 (5) | 0.091 (5) | 0.150 (7) | −0.004 (4) | 0.027 (5) | −0.002 (5) |
| C25 | 0.119 (7) | 0.102 (6) | 0.170 (9) | −0.009 (5) | −0.022 (6) | −0.002 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C12 | 1.665 (5) | C10—C11 | 1.418 (6) |
| Cl1—C7 | 1.717 (5) | C14—C15 | 1.481 (8) |
| Cl2—C25 | 1.725 (7) | C14—H14A | 0.9700 |
| F1—C8 | 1.341 (6) | C14—H14B | 0.9700 |
| O1—C11 | 1.231 (5) | C15—H15A | 0.9600 |
| O2—C13 | 1.229 (5) | C15—H15B | 0.9600 |
| O3—C21 | 1.208 (5) | C15—H15C | 0.9600 |
| O4—C21 | 1.340 (5) | C16—C17 | 1.479 (8) |
| O4—C22 | 1.451 (6) | C16—H16A | 0.9700 |
| N1—C5 | 1.326 (5) | C16—H16B | 0.9700 |
| N1—C1 | 1.375 (5) | C17—H17A | 0.9600 |
| N1—C18 | 1.458 (5) | C17—H17B | 0.9600 |
| N2—C12 | 1.352 (6) | C17—H17C | 0.9600 |
| N2—C11 | 1.429 (5) | C18—C20 | 1.469 (6) |
| N2—C14 | 1.491 (6) | C18—C19 | 1.479 (6) |
| N3—C12 | 1.379 (6) | C18—H18A | 0.9800 |
| N3—C13 | 1.406 (5) | C19—C20 | 1.462 (7) |
| N3—C16 | 1.477 (6) | C19—H19A | 0.9700 |
| C1—C6 | 1.409 (6) | C19—H19B | 0.9700 |
| C1—C2 | 1.415 (5) | C20—H20A | 0.9700 |

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| C2—C9 | 1.404 (6) | C20—H20B | 0.9700 |
| C2—C3 | 1.441 (5) | C22—C23 | 1.402 (9) |
| C3—C4 | 1.391 (6) | C22—H22A | 0.9700 |
| C3—C10 | 1.449 (5) | C22—H22B | 0.9700 |
| C4—C5 | 1.390 (5) | C23—C24 | 1.484 (10) |
| C4—C21 | 1.486 (6) | C23—H23A | 0.9700 |
| C5—H5A | 0.9300 | C23—H23B | 0.9700 |
| C6—C7 | 1.361 (7) | C24—C25 | 1.544 (10) |
| C6—H6A | 0.9300 | C24—H24A | 0.9700 |
| C7—C8 | 1.396 (7) | C24—H24B | 0.9700 |
| C8—C9 | 1.348 (7) | C25—H25A | 0.9700 |
| C9—H9A | 0.9300 | C25—H25B | 0.9700 |
| C10—C13 | 1.406 (6) | | |

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| C21—O4—C22 | 116.5 (4) | H15A—C15—H15C | 109.5 |
| C5—N1—C1 | 118.9 (3) | H15B—C15—H15C | 109.5 |
| C5—N1—C18 | 119.8 (3) | N3—C16—C17 | 111.4 (5) |
| C1—N1—C18 | 121.3 (3) | N3—C16—H16A | 109.4 |
| C12—N2—C11 | 124.5 (4) | C17—C16—H16A | 109.4 |
| C12—N2—C14 | 120.0 (4) | N3—C16—H16B | 109.4 |
| C11—N2—C14 | 115.5 (4) | C17—C16—H16B | 109.4 |
| C12—N3—C13 | 124.3 (4) | H16A—C16—H16B | 108.0 |
| C12—N3—C16 | 119.1 (4) | C16—C17—H17A | 109.5 |
| C13—N3—C16 | 116.6 (4) | C16—C17—H17B | 109.5 |
| N1—C1—C6 | 119.4 (4) | H17A—C17—H17B | 109.5 |
| N1—C1—C2 | 119.8 (4) | C16—C17—H17C | 109.5 |
| C6—C1—C2 | 120.8 (4) | H17A—C17—H17C | 109.5 |
| C9—C2—C1 | 117.2 (4) | H17B—C17—H17C | 109.5 |
| C9—C2—C3 | 122.0 (4) | N1—C18—C20 | 119.5 (4) |
| C1—C2—C3 | 120.8 (4) | N1—C18—C19 | 118.6 (4) |
| C4—C3—C2 | 115.8 (3) | C20—C18—C19 | 59.5 (3) |
| C4—C3—C10 | 122.8 (4) | N1—C18—H18A | 115.9 |
| C2—C3—C10 | 121.3 (4) | C20—C18—H18A | 115.9 |
| C5—C4—C3 | 120.2 (4) | C19—C18—H18A | 115.9 |
| C5—C4—C21 | 116.0 (4) | C20—C19—C18 | 59.9 (3) |
| C3—C4—C21 | 123.3 (4) | C20—C19—H19A | 117.8 |
| N1—C5—C4 | 124.1 (4) | C18—C19—H19A | 117.8 |
| N1—C5—H5A | 117.9 | C20—C19—H19B | 117.8 |
| C4—C5—H5A | 117.9 | C18—C19—H19B | 117.8 |
| C7—C6—C1 | 119.6 (4) | H19A—C19—H19B | 114.9 |
| C7—C6—H6A | 120.2 | C19—C20—C18 | 60.6 (3) |
| C1—C6—H6A | 120.2 | C19—C20—H20A | 117.7 |
| C6—C7—C8 | 119.6 (5) | C18—C20—H20A | 117.7 |
| C6—C7—Cl1 | 120.5 (4) | C19—C20—H20B | 117.7 |
| C8—C7—Cl1 | 119.9 (4) | C18—C20—H20B | 117.7 |
| F1—C8—C9 | 119.8 (5) | H20A—C20—H20B | 114.8 |
| F1—C8—C7 | 118.3 (5) | O3—C21—O4 | 125.1 (4) |
| C9—C8—C7 | 121.9 (5) | O3—C21—C4 | 123.4 (4) |
| C8—C9—C2 | 120.9 (4) | O4—C21—C4 | 111.3 (4) |

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| C8—C9—H9A | 119.6 | C23—C22—O4 | 113.5 (6) |
| C2—C9—H9A | 119.6 | C23—C22—H22A | 108.9 |
| C13—C10—C11 | 119.8 (4) | O4—C22—H22A | 108.9 |
| C13—C10—C3 | 119.5 (4) | C23—C22—H22B | 108.9 |
| C11—C10—C3 | 120.5 (4) | O4—C22—H22B | 108.9 |
| O1—C11—C10 | 125.3 (4) | H22A—C22—H22B | 107.7 |
| O1—C11—N2 | 118.0 (4) | C22—C23—C24 | 117.0 (8) |
| C10—C11—N2 | 116.8 (4) | C22—C23—H23A | 108.1 |
| N2—C12—N3 | 116.2 (4) | C24—C23—H23A | 108.1 |
| N2—C12—S1 | 122.2 (4) | C22—C23—H23B | 108.1 |
| N3—C12—S1 | 121.7 (4) | C24—C23—H23B | 108.1 |
| O2—C13—N3 | 117.9 (4) | H23A—C23—H23B | 107.3 |
| O2—C13—C10 | 124.4 (4) | C23—C24—C25 | 112.8 (7) |
| N3—C13—C10 | 117.8 (4) | C23—C24—H24A | 109.0 |
| C15—C14—N2 | 112.8 (4) | C25—C24—H24A | 109.0 |
| C15—C14—H14A | 109.0 | C23—C24—H24B | 109.0 |
| N2—C14—H14A | 109.0 | C25—C24—H24B | 109.0 |
| C15—C14—H14B | 109.0 | H24A—C24—H24B | 107.8 |
| N2—C14—H14B | 109.0 | C24—C25—Cl2 | 113.3 (6) |
| H14A—C14—H14B | 107.8 | C24—C25—H25A | 108.9 |
| C14—C15—H15A | 109.5 | Cl2—C25—H25A | 108.9 |
| C14—C15—H15B | 109.5 | C24—C25—H25B | 108.9 |
| H15A—C15—H15B | 109.5 | Cl2—C25—H25B | 108.9 |
| C14—C15—H15C | 109.5 | H25A—C25—H25B | 107.7 |

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| C5—N1—C1—C6 | 175.2 (4) | C12—N2—C11—O1 | 171.1 (5) |
| C18—N1—C1—C6 | −2.4 (6) | C14—N2—C11—O1 | −8.3 (6) |
| C5—N1—C1—C2 | −3.3 (6) | C12—N2—C11—C10 | −7.7 (6) |
| C18—N1—C1—C2 | 179.1 (4) | C14—N2—C11—C10 | 173.0 (4) |
| N1—C1—C2—C9 | 175.0 (4) | C11—N2—C12—N3 | 1.1 (7) |
| C6—C1—C2—C9 | −3.5 (6) | C14—N2—C12—N3 | −179.6 (4) |
| N1—C1—C2—C3 | −1.8 (6) | C11—N2—C12—S1 | −177.9 (3) |
| C6—C1—C2—C3 | 179.7 (4) | C14—N2—C12—S1 | 1.4 (6) |
| C9—C2—C3—C4 | −171.0 (4) | C13—N3—C12—N2 | 4.1 (7) |
| C1—C2—C3—C4 | 5.6 (6) | C16—N3—C12—N2 | −175.3 (4) |
| C9—C2—C3—C10 | 7.2 (6) | C13—N3—C12—S1 | −176.8 (4) |
| C1—C2—C3—C10 | −176.3 (4) | C16—N3—C12—S1 | 3.7 (7) |
| C2—C3—C4—C5 | −4.6 (6) | C12—N3—C13—O2 | 177.8 (4) |
| C10—C3—C4—C5 | 177.3 (4) | C16—N3—C13—O2 | −2.7 (6) |
| C2—C3—C4—C21 | −176.7 (4) | C12—N3—C13—C10 | −2.3 (7) |
| C10—C3—C4—C21 | 5.2 (6) | C16—N3—C13—C10 | 177.1 (4) |
| C1—N1—C5—C4 | 4.5 (6) | C11—C10—C13—O2 | 175.2 (4) |
| C18—N1—C5—C4 | −177.9 (4) | C3—C10—C13—O2 | −1.3 (7) |
| C3—C4—C5—N1 | −0.3 (6) | C11—C10—C13—N3 | −4.7 (6) |
| C21—C4—C5—N1 | 172.3 (4) | C3—C10—C13—N3 | 178.8 (4) |
| N1—C1—C6—C7 | −176.7 (5) | C12—N2—C14—C15 | −87.5 (6) |
| C2—C1—C6—C7 | 1.8 (7) | C11—N2—C14—C15 | 91.9 (6) |
| C1—C6—C7—C8 | −0.2 (9) | C12—N3—C16—C17 | 92.7 (6) |
| C1—C6—C7—Cl1 | 178.2 (4) | C13—N3—C16—C17 | −86.8 (6) |

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| C6—C7—C8—F1 | 179.6 (5) | C5—N1—C18—C20 | 117.2 (5) |
| Cl1—C7—C8—F1 | 1.2 (8) | C1—N1—C18—C20 | −65.2 (5) |
| C6—C7—C8—C9 | 0.5 (10) | C5—N1—C18—C19 | 48.1 (6) |
| Cl1—C7—C8—C9 | −178.0 (5) | C1—N1—C18—C19 | −134.3 (5) |
| F1—C8—C9—C2 | 178.6 (5) | N1—C18—C19—C20 | 109.3 (5) |
| C7—C8—C9—C2 | −2.3 (9) | N1—C18—C20—C19 | −107.7 (4) |
| C1—C2—C9—C8 | 3.8 (7) | C22—O4—C21—O3 | −8.1 (7) |
| C3—C2—C9—C8 | −179.5 (5) | C22—O4—C21—C4 | 176.5 (4) |
| C4—C3—C10—C13 | 43.7 (6) | C5—C4—C21—O3 | −123.8 (5) |
| C2—C3—C10—C13 | −134.2 (4) | C3—C4—C21—O3 | 48.6 (7) |
| C4—C3—C10—C11 | −132.7 (5) | C5—C4—C21—O4 | 51.7 (5) |
| C2—C3—C10—C11 | 49.3 (6) | C3—C4—C21—O4 | −135.9 (4) |
| C13—C10—C11—O1 | −169.4 (5) | C21—O4—C22—C23 | 102.8 (7) |
| C3—C10—C11—O1 | 7.0 (7) | O4—C22—C23—C24 | 55.7 (10) |
| C13—C10—C11—N2 | 9.3 (6) | C22—C23—C24—C25 | 178.9 (7) |
| C3—C10—C11—N2 | −174.3 (4) | C23—C24—C25—Cl2 | −76.6 (9) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C18—H18*A*···O2$^{i}$ | 0.98 | 2.32 | 3.014 (6) | 127 |
| C20—H20*A*···O3$^{ii}$ | 0.97 | 2.56 | 3.233 (6) | 126 |
| C25—H25*A*···F1$^{iii}$ | 0.97 | 2.51 | 3.371 (10) | 148 |

Symmetry codes: (i) −*x*, *y*, −*z*+1/2; (ii) −*x*, −*y*+1, −*z*; (iii) *x*, *y*+1, *z*.